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The Effect of Ageing on
PBXW-115 (Aust.), PBXN-103
and PBXN-105

G. Bocksteiner and D.J. Whelan

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ABSTRACT

When PBXW-115 (Aust.) is exposed to accelerated ageing according to the program set out in DEFAUST 5168: Cycle B2, mass loss (attributed to loss of plasticiser, isodecyl pelargonate) occurs, the Shore A hardness increases and the tensile properties alter somewhat.

There is a link between these changes and it is probably related to the plasticiser content of the samples. From a kinetic analysis, it appears as if there may be two consecutive mass loss processes operating in the accelerated ageing of PBXW-115 and each obeys a first order reaction kinetics profile; the first predominates up to the stage where a mass loss equivalent to ca. 55 % of plasticiser occurs, the second operates where the plasticiser content is low. However, in the time domain studied (one year), there is no evidence of catastrophic structural change (loss of integrity or propensity to crumble) in PBXW-115 (Aust.) under worst case conditions (test pieces of small volume and large relative surface area), suggesting that the explosive charge in warheads filled with PBXW-115 (Aust.) should remain intact over a long interval of time.

The tensile properties of PBXW-115 (Aust.) are similar to that of its US counterpart and not dissimilar to those of PBXN-103, PBXN-105 and PBXN-106; all are rubber-like materials, with lower relative tensile strengths at 20-25°C (σ_m, maximum stress values of 0.3 to 1.1 MPa), higher relative extensibilities (ε_m, strains at maximum load of 8 -15%) and very low tangential moduli (E, 5-20 MPa), compared to those of Composition B and H-6 (σ_m 1-3 MPa, ε_m 0.02%, E ca.12000 MPa).

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The Effect of Ageing on PBXW-115 (Aust.), PBXN-103 and PBXN-105

Executive Summary

PBXW-115 is a polymer bonded explosive (PBX) which was developed at Naval Surface Warfare Center, White Oak, Silver Spring, MD, USA in the late 70s / early 80s. Since that time, PBXW-115 has been fully qualified by the US Navy, renamed PBXN-111 and accepted for use as a mine demolition charge, Mk 98 MNC, which will conform to Insensitive Munitions requirements.

More recently, an Australian version of PBXW-115, designated PBXW-115 (Aust.), has also been developed and several reports on its explosive properties and performance have been published from this laboratory.

This present report studies the effect of accelerated ageing on laboratory samples of PBXW-115 (Aust.) in terms of changes in the surface hardness, mass loss, and tensile properties with time. It has been found that:

1) the Shore A hardness increases on ageing, probably due to progressive loss of plasticiser,
2) there is also a loss of mass, which follows an apparent first-order reaction for up to 55 % plasticiser loss, and
3) the tensile properties are also seen to alter with time.

Similar results occur with aged samples of PBXN-103, the explosive warhead fill in the RAN Mk 46 torpedo, and PBXN-105, the warhead fill in the RAN Mk 48 torpedo.

Even in the worst-case scenario, small samples of all of these materials retain their rubber-like qualities, after exposure to accelerated ageing (PBXW-115 (Aust.): Cycle B2 Ageing, 12 months; PBXN-103: 60°C, 3 months; PBXN-105: 60°C, 6 months). None of them show any evidence of catastrophic structural change (deconsolidation, loss of integrity or propensity to crumble). This suggests that the explosive charge in warheads filled with these materials in bulk should remain intact over a long interval of time.

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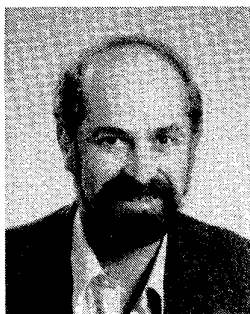
Gunter Bocksteiner graduated from Footscray Institute of Technology in Applied Chemistry and joined MRL in 1968. He has worked widely within MRL, initially on the mechanistic chemistry of chemiluminescent materials, then biologically active surface coatings and the effects of the marine environment on defence materiel. In 1980 he commenced work on determination of explosives hazard, and lately has been working on polymer bonded explosives formulations.

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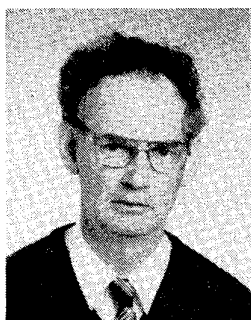
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Contents

1. Introduction

PBXW-115 is a polymer bonded explosive (PBX) which was developed at Naval Surface Warfare Center, White Oak, Silver Spring, MD, USA in the late 70s / early 80s, for potential use as an insensitive explosive fill for underwater applications [1]. Since that time, PBXW-115 has been fully qualified, accepted as a possible IM warhead fill by the US Navy and renamed PBXN-111 [2].

More recently, an Australian version of PBXW-115, which the authors have designated PBXW-115 (Aust.), has also been developed [3] and reports on its explosive properties and performance have either been published or are in the process of publication [4-7]. This present report studies the effect of accelerated ageing on laboratory samples of PBXW-115 (Aust.) in terms of changes in the surface hardness, mass loss, and tensile properties with time. In addition, the authors have taken the opportunity to place these results in some perspective by comparing the results of tensile testing on aged PBXW-115 (Aust.) with those reported for aged samples of PBXN-103 and PBXN-105, two underwater warhead fills in the present RAN inventory, and for PBXN-106, whose composition is very similar to that of PBXN-105.

2. Formulations

2.1 PBXW-115 (Aust.) and PBXN-111

Both PBXW-115 (Aust) and its American counterpart, PBXN-111, are cast-cured PBX compositions, prepared from RDX (bimodal), Al powder, ammonium perchlorate and a polyurethane binder.

Their nominal compositions [1, 3] are the same, namely:-

Ingredient	Function	Mass (%)
Al powder	Metal fuel	25
Ammonium perchlorate (AP)	Oxidant	43
RDX, Class 1	High explosive	12
RDX, Class 5	High explosive	8
Isodecyl pelargonate	Plasticiser & fuel	5.7
HTPB / IPDI polyurethane, cross-linked and cured *in situ* at 60°C.	Binder & fuel	6.3

In practice, however, significant differences in the critical diameters and shock sensitivities of the two formulations have been observed [1,4] and these have been attributed to differences in the RDX used in the two formulations [2,5].

Both formulations have been qualified for service applications.

Just recently, the Australian Ordnance Council announced that PBXW-115 (Aust.) had been qualified for use as a main charge fill for large underwater blast weapons [8] and it is known that PBXN-111 has been qualified by the US Navy for use as the mine neutralisation charge designated Mk 98 MNC [9].

The authors were recently informed [10] that three different formulations closely related to PBXN-111 have been either developed or introduced into service in Europe since 1991. Great Britain has developed one formulation for underwater applications and they call their formulation ROWANEX 1301, the name being an acronym for Royal Ordnance Waltham Abbey Naval Explosive [10]. There is also a German equivalent, KS 57, which has been developed by Messerschmitt-Bölkow-Blohm (MBB) GmbH, and is made from bimodal RDX (24%) / Al (24%) / AP (40%) / plasticised HTPB binder (12%) [10, 11], and two SNPE (France) PBXN-111 - styled fills, called B2211B and B2211D, which have been incorporated in the warheads of the current versions of the UK-based Marconi mine, Stonefish [10,12].

2.2 PBXN-103

PBXN-103, being the warhead fill in the RAN Mk 46 torpedo, was the first polymer-bonded explosive to be used in an RAN warhead. PBXN-103 has also been widely used in a variety of other underwater warheads in the USN inventory, including the Mk 46 torpedo, the earlier versions of the Mk 48 torpedo, the Captor (Encapsulated Torpedo) Mine and the Sabre Mk 57 Destructor [9, 12]. Although it was introduced into service over twenty years ago, it is still the most powerful and damaging of the underwater fills currently in use in the USN and its performance provides the benchmark against which other underwater explosives are compared.

Its composition [13, 14] is as follows:-

Ingredient	Function	Mass (%)
Ammonium perchlorate	Explosive Oxidant	40
Al powder	Fuel	27
Trimethylolethane trinitrate, TMETN	Energetic Plasticiser	23
Triethyleneglycol dinitrate, TEGDN	Energetic Plasticiser	2.5
Pelletised Nitrocellulose	Binder	6
Ethyl Centralite	Stabilizer (NO_x scavenger)	1.3
Resorcinol	Antioxidant	0.2

Unfortunately, there are several instances where current munitions filled with PBXN-103 do not satisfy IM criteria. As a result, some new programs have been put in place over the last decade to find a replacement underwater warhead fill which will conform to Insensitive Munitions (IM) guidelines and still retain or exceed the performance capability of PBXN-103. According to US sources [9], PBXN-111 does not have quite the same underwater energy output as PBXN-103 although it is better credentialled in as far as compliance to IM guidelines is concerned.

2.3 PBXN-105 and PBXN-106

PBXN-105 is the warhead fill in the RAN Mk 48 torpedo; in terms of energy output, it is very similar to PBXN-103. It was originally formulated to yield a very dense RDX-containing explosive of high underwater shock energy and high bubble energy.

Its composition [13, 15] is given below:-

Ingredient	Function	Mass (%)
Ammonium perchlorate	Explosive Oxidant	49.8
Al powder	Fuel	25.8
RDX	High Explosive	7
Bis (dinitro- propyl acetal / formal), BDNPA/F	Energetic plasticiser	12.9
Polyoxyethylene glycol (PEG) / trimethylolpropane (TMP)/ TDI-crosslinked polyurethane	Binder	4.3
Phenyl β-naphthylamine	Antioxidant	0.2

PBXN-105 was considered, in the late 80s, as a possible fill for the UK Spearfish torpedo but was subsequently passed over, presumably in favour of another (unspecified) underwater explosive. PBXN-105 is closely related to the US propellant formulation, ANP 2969, which consists of AP (55%), Al (20%), BDNPA/F (12.5%), PEG/TMP/TDI binder (12.5%) and is used in one of the stages of the Polaris launch vehicle.

PBXN-106 is an RDX-based PBX [13,15]. It is closely related to PBXN-105, being made up of RDX (Class 1, 60 %; Class 5, 15 %) in a PEG/TMP/IPDI polyurethane binder (6.5%) and heavily plasticised with BDNPA/F (18.5%). It has tensile properties similar to PBXN-105 and is included in this report as a comparative reference material.

3. Experimental

3.1 Samples of PBXW-115 (Aust.)

PBXW-115 (Aust.), like its USN counterpart, PBXN-111, is vacuum-cast at ambient temperature and cured at 60°C [3].

Samples for ageing studies were prepared with particular ends in mind.

a. Change in Hardness with Time at 60°C.

Cylindrical samples, 80 mm in diameter and 300 mm in length, were prepared in order to study the change in hardness of the PBX with time and depth of sample at 60°C. Although the results of this study have already been published [3], points of interest specifically relating to this report will be presented, where appropriate.

b. Change in Mass under Accelerated Ageing Conditions.

Two cylindrical samples of different dimensions were prepared for mass loss studies; one sample had a mass approx. 72 g (actual: 72.85 g), the other had a mass approx. one-half the first sample, 36 g (actual: 37.92 g). They were chosen from available material to ascertain how materials of different size and cross sectional area behave under accelerated ageing conditions (as described in Sect. 3.2 and Table 1).

c. Change in Tensile Properties under Accelerated Ageing Conditions.

PBXW-115 (Aust.) for tensile testing was cast to a thickness of approx. 7 mm onto rectangular trays (300 mm x 450 mm) lined with a film of mylar and then extensively degassed under vacuum (10^{-2} -10^{-3} torr). A second film of mylar was then placed over this degassed material and the top film rolled to smooth its surface and the material was placed in an oven for curing at 60°C.

Some of this cured material was placed in an environmental test chamber (see below) while other material designated for long term storage at ambient laboratory conditions was placed in an air-tight closed vessel and stored appropriately. Samples were taken out as required; the mylar sheets were then peeled off and the dogbone-shaped tensile test specimens, approximately 40 mm^2 cross sectional area, were cut out immediately prior to testing.

3.2 Accelerated Ageing

Various samples of PBXW-115 (Aust.), set aside for studying the effect of accelerated ageing on either the tensile properties or mass loss, were placed in the one of the

environmental test chambers, located within Weapons Systems Division, AMRL, Maribyrnong. The chamber was programmed to operate on a recurring 24 hour cycle timetable described in Cycle B2: DEF (AUST) 5168 [16]. Details of this program are summarised in Table 1.

This accelerated ageing program was chosen in consultation with Australian Ordnance Council guidelines; the program represented a very severe but appropriate upper temperature/humidity regime to which stores filled with PBXW-115 (Aust.) could be expected to survive for a reasonable time.

Table 1: The Accelerated Ageing Regime corresponding to Simulated Storage Conditions : Cycle B2 , DEF(AUST) 5168 [16].

Time, hr.	Temperature, °C	Relative Humidity, %
0000	33	68
0300	32	71
0600	31	75
0900	42	43
1200	57	22
1500	63	19
1800	57	22
2100	38	54
2400	33	68

Over a twenty-four hour cycle carried out under this B2 regime, the time-averaged temperature in the chamber can be considered 44°C corresponding to a time-averaged relative humidity of 37 %; under these conditions, one can estimate that there would be an approximate 4.5-fold rate enhancement factor (relative to that for material at 25°C) observable for material transport processes (assuming the process under investigation has an Arrhenius activation energy of 60 kJ mol^{-1}, a typical activation energy for diffusion processes involving the bleeding of plasticisers in rubbers and paints, [17]).

Samples were removed after exposure of approximately 3 months (actual: 85 days), 6 months (actual: 193 days) and 12 months (actual: 352 days).

3.3 Shore-A Hardness

Sample hardness was carried out according to ASTM procedures, as presented in ASTM Standard D2240 [18].

Detailed results for samples stored at 60°C for periods up to 60 days have been reported previously [3, pp 20-21, 40-43].

Measurements were performed at 20 - 24°C using a Shore A-2 Durometer with a Shore Conveloader test stand employing a hydraulic cylinder to control the rate of application of the standard 1 kg indentor to the sample.

3.4 Tensile Testing

Tensile testing was carried out using a Lloyd Mechanical Test Apparatus Model LR50K, manufactured by Lloyd Instrument Ltd., Hampshire, England, to the specifications covered by the American Society for the Testing of Materials, Method ASTM D 412, Type C [18b]; the instrument operated at a constant strain rate of 50 mm min^{-1}.

4. Results

4.1 Shore-A Hardness of PBXW-115 (Aust.)

It was found that the Shore A hardness of discs of PBXW-115 (Aust.) samples investigated in this study did increase with time.

For fresh samples, the hardness of the top surface was found to be 60 ± 2 Shore-A units (10 measurements). On ageing at 60°C, this increased to 64 ± 3 Shore-A units (top surface, 10 measurements, 45 days), probably reflecting the loss of plasticiser through diffusion and evaporation.

The Shore A hardness of unplasticised HTPB-based propellants also increases on ageing [19], the change, in the first instance, probably being attributable to the occurrence of increased crosslinking as time progresses.

The Shore-A hardness of fresh PBXN-111 has been reported to be 53 Shore-A units [1], a value lower but, perhaps, not far removed from that of PBXW-115 (Aust.).

4.2 Mass Loss in PBXW-115 (Aust.)

Two cylinders of PBXW-115 (Aust.), one of mass 72.85 g (height 33.8 mm and diameter 39.1 mm), the other of mass 37.92 g (height 24.4 mm and diameter 33.2 mm) were placed on a tray in the environmental test chamber and exposed to the B2 Accelerated Ageing Regime, for 6 months (193 days). They were weighed periodically throughout the ageing process (Table 2) and the dimensions measured at the end of the accelerated ageing period.

The dimensions of the two cylinders did not alter perceptibility during the six month ageing process.

In each sample, there appear to be two mass loss processes operating and these are analysed more fully in the Discussion. One assumes that the mass loss is due to the loss of plasticiser in the PBX and, if this is so, then one can estimate the zero-time plasticiser content in the ageing samples, using the data derived from Table 2.

From Sample A, a simple plot of mass loss vs. time, out to the full 193 days, can be fitted to a quadratic equation,

$$y(A) = 0.06739 + 0.025499\,t - 6.154 \times 10^{-5}\,t^2,$$
with a coefficient of determination of 0.9986,

where y (A) is the mass loss (in g) from Sample A and t is the time that the sample spent (in days) in the environmental chamber.

The same data can also be fitted to a cubic relationship,

$$y(A) = 0.0449 + 0.02727\,t - 8.5450 \times 10^{-5}\,t^2 + 8.2860 \times 10^{-8}\,t^3,$$
with a coefficient of determination of 0.9989,

but, as with the quadratic relationship, there is no real physical meaning to be attached to the line fit, especially at high values of t (perhaps t >100).

However, at zero time, one may, perhaps, assume that the apparent mass loss at zero time can be taken as an average of the two values obtained from the above relationships, viz. 0.05_6 ($\pm$ 0.00_9), and that this value can be applied as a correction to the plasticiser content of Sample A. The plasticiser content of Sample A is calculated on the basis of formulation data (4.15 g, 5.7 %) and, by applying the correction above, this gives a corrected plasticiser content of Sample A of 4.09 g.

From Sample B, the mass loss / time data can be fitted to similar equations of the same form, namely:-

$$y(B) = 0.1546 + 0.01883\,t - 5.931 \times 10^{-5}\,t^2,$$
with a coefficient of determination of 0.9734, and

$$y(B) = 0.0292 + 0.02871\,t - 1.9265 \times 10^{-4}\,t^2 + 4.6207 \times 10^{-7}\,t^3,$$
with a coefficient of determination of 0.9967,

where y (B) is the mass loss (in g) and t is the time that the sample spent (in days) in the environmental chamber.

Again, at zero time, one assumes that the apparent mass loss at zero time can be taken as an average of the two values obtained from the above relationships, viz. 0.09_2 ($\pm$ 0.06_3). This value is then applied as a correction to the plasticiser content of Sample B calculated on the basis of formulation data (2.16g, 5.7 %), yielding a corrected plasticiser content of 2.07 g at the start of the accelerated ageing process for Sample B.

Table 2: The Loss of Mass in Samples of PBXW-115 (Aust.) exposed to Accelerated Ageing : Cycle B2 with Time.

Time (days)	Sample A Original Mass: 72.85 g Mass loss (g)	Sample B Original Mass: 37.92 g Mass loss (g)
0	0	0
15	0.50	0.43
28	0.77	0.71
49	1.15	1.08
63	1.42	1.19
77	1.67	1.28
91	1.87	1.36
116	2.23	1.47
126	2.29	1.49
140	2.42	1.55
157	2.54	1.62
169	2.60	1.64
176	2.64	1.64
193	2.73	1.68

Sample A: a cylinder of height 33.8 mm and 39.1 mm diameter.
Sample B: a cylinder of height 24.4 mm and 33.2 mm diameter.

4.3 Tensile Testing of PBXW-115 (Aust.)

The results of tensile testing of dumbbell shaped test samples of PBXW-115 (Aust.) stored for periods up to one year are recorded in Tables 3 (Room Temperature) and 4 (Room Temperature and Accelerated Ageing).

The stress / strain profiles are similar to those obtained from many rubber-like materials [20, 21].

From the data summarised in Tables 3 and 4, it is apparent that the tensile properties of the samples tested after 12 weeks (85 days) under both ambient conditions and accelerated ageing conditions are lower than those from freshly prepared samples and from those aged out to six months (193 days). It is apparent that changes are occurring

in the early stages of the ageing process (during the first 12 weeks) which affect the tensile properties in a manner different to those changes occurring over the ensuing three months.

Table 3: Tensile Testing of PBXW-115 (Aust.), stored at Room Temperatures in an Air-tight Container, for Periods up to One Year.

Sample	Cross-Sectional Area, (mm^2)	Maximum. Load, (N)	Maximum Stress, (MPa)	Extension at Maximum. Load (mm)	Strain at Max. Load (%)	Stress at Break (MPa)	Strain at Break (%)	Modulus of Elasticity (MPa)
Fresh Sample, cured 7 days earlier: 8 Samples tested								
Parameter	39.57	19.14	0.484	3.22	9.76	0.457	10.5	6.18
Std. Devn.	0.68	0.48	0.013	0.43	1.31	0.021	1.5	0.48
Compare: PBXW-115 (USA) / N-111 (Anderson and Leahy, 1985, [1])			0.579		8.36			approx. 7
Aged Sample, 85 days. 8 Samples tested								
Parameter	38.23	19.60	0.513	2.90	8.78	0.467	9.69	7.77
Std. Devn.	0.52	0.53	0.018	0.49	1.48	0.015	1.72	1.14
Aged Sample, 6 months. 8 Samples tested.								
Parameter	38.54	23.15	0.600	3.58	10.86	not measured		7.42
Std. Devn.	0.59	2.06	0.044	0.19	0.58			0.73
Aged Sample, 50 weeks. 8 Samples tested.								
Parameter	38.71	24.04	0.621	3.48	10.66	0.561	11.54	7.26
Std. Devn.	0.55	1.17	0.022	0.35	1.05	0.033	1.78	0.87

Table 4: Tensile Testing of PBXW-115 (Aust.), either stored at Room Temperatures in an Air-tight Container or subjected to the Accelerated Ageing Regime, Cycle B2, for Periods up to One Year.

Sample	Cross Sectional Area, (mm^2)	Maximum Load, (N)	Maximum Stress, (MPa)	Extension at Maximum Load (mm)	Strain at Max. Load (%)	Stress at Break, (MPa)	Strain at Break, (%)	Modulus of Elasticity, (MPa)
Fresh Sample, cured 7 days earlier: 8 Samples tested								
Parameter	39.57	19.14	0.484	3.22	9.76	0.457	10.5	6.18
Std. Devn.	0.68	0.48	0.013	0.43	1.31	0.021	1.5	0.48
Aged Sample, 85 days at Room Temperature in an Air-tight Container. 8 Samples tested								
Parameter	38.23	19.60	0.513	2.90	8.78	0.467	9.69	7.77
Std. Devn.	0.52	0.53	0.018	0.49	1.48	0.015	1.72	1.14
Aged Sample, 85 days Accelerated Ageing, Cycle B2. 8 Samples tested.								
Parameter	38.34	18.02	0.473	2.82	8.53	0.435	9.43	7.21
Std. Devn.	0.72	0.56	0.014	0.23	0.68	0.020	0.86	0.39
Aged Sample, 6 months at Room Temperature in an Air-tight Container. 8 Samples tested.								
Parameter	38.54	23.15	0.600	3.58	10.86	not measured		7.42
Std. Devn.	0.59	2.06	0.04	0.19	0.58			0.73
Aged Sample, 6 months Accelerated Ageing, Cycle B2. 8 Samples tested.								
Parameter	38.27	20.92	0.536	3.08	9.23	0.493	9.61	7.66
Std. Devn.	0.33	1.27	0.040	0.20	0.61	0.021	0.70	0.47
Aged Sample, 50 weeks at Room Temperature in an Air-tight Container. 8 Samples tested.								
Parameter	38.71	24.04	0.621	3.48	10.66	0.561	11.54	7.26
Std. Devn.	0.55	1.17	0.022	0.35	1.05	0.033	1.78	0.87
Aged Sample, 50 weeks Accelerated Ageing, Cycle B2. 8 Samples tested.								
Parameter	38.62	21.36	0.553	3.13	9.55	0.511	10.03	8.15
Std. Devn.	0.47	1.02	0.026	0.38	0.84	0.022	1.32	0.72

5. Discussion: The Accelerated Ageing of PBXW-115 (Aust.)

5.1 Changes in Mass and Hardness of PBXW-115 (Aust.)

In Table 2, the experimental data associated with the loss of mass from the two samples of PBXW-115 (Aust.) has already been presented.

If one assumes that the mass loss is due solely to the loss of plasticiser, isodecyl pelargonate (IDP), that plasticiser has been lost in the manufacturing and curing stages and that the amount of plasticiser in each sample at zero time corresponds to that proposed in Section 4.2, then the data in Table 2 can be represented in the form of Table 5. Here, the fractional loss of mass, F, is defined as the ratio of mass loss to the (corrected) mass of plasticiser in the original sample; by definition, (1 - F) is then the fractional ratio of the mass of plasticiser remaining in the sample.

Table 5: *Change in Mass with time for the Two Samples of PBXW-115 (Aust.)*

	Sample A		Sample B	
	Original mass: 72.85 g		Original mass: 37.92 g	
	Mass of Plasticiser: 4.09 g (corr.)		Mass of Plasticiser: 2.07 g (corr.)	
Time (days)	Fractional loss of Plasticiser, F	Fraction of Plasticiser remaining, (1-F)	Fractional loss of Plasticiser, F	Fraction of Plasticiser remaining, (1-F)
0	0	1	0	1
15	0.123	0.877	0.208	0.792
28	0.189	0.811	0.343	0.657
49	0.281	0.719	0.522	0.478
63	0.345	0.653	0.575	0.425
77	0.409	0.591	0.618	0.382
91	0.457	0.543	0.657	0.343
116	0.545	0.455	0.710	0.290
126	0.569	0.440	0.720	0.280
140	0.592	0.408	0.749	0.251
157	0.624	0.376	0.783	0.217
169	0.636	0.364	0.792	0.208
176	0.646	0.354	0.792	0.208
193	0.667	0.333	0.812	0.188

If one assumes that the mass losses follow first order kinetics, i.e. the time rate of loss of mass is proportional to the mass of plasticiser remaining in the PBX matrix, then it can be seen from plots of ln (1-F) vs. time (t, days) that two consecutive processes are occurring.

For Sample A, it appears that, over the first 116 days under accelerated ageing conditions (at which time 55 % of the plasticiser has disappeared), the predominant mass-loss reaction follows a rate law

$$\ln (1\text{-}F) = -0.00618 \ t \qquad\qquad [\ Eq.\ 1A]$$

with a linear least squares coefficient of determination, r^2 equal to 0.9959,

while, beyond 140 days, a slower reaction clearly predominates; this latter reaction follows the rate law

$$\ln (1-F) = -0.00377\, t - 0.3742 \qquad \text{[Eq. 2A]}$$

with a linear least squares coefficient of determination, r^2, equal to 0.9905.

For the smaller sample, Sample B, if one plots out the data from Table 5, it can be seen that, over the first 77 days under accelerated ageing conditions (at which time 62 % of the plasticiser has disappeared), the predominant mass-loss reaction follows a rate law

$$\ln (1-F) = -0.0135\, t \qquad \text{[Eq. 1B]}$$

with a linear least squares coefficient of determination, r^2 equal to 0.980,

while, beyond 140 days (at which time 75 % of the plasticiser has disappeared), the following mass-loss reaction predominates,

$$\ln (1-F) = -0.00511\, t - 0.6906 \qquad \text{[Eq. 2B]}$$

with a linear least squares coefficient of determination, r^2, equal to 0.945.

In the time domain from 77 days to 140 days, the reaction kinetics manifest themselves in a less well-defined composite profile.

Overall, it is apparent that, under the accelerated ageing regime, there are two mass-loss processes operating and these are kinetically first-order. In the two examples studied here, the initial process predominates up to the stage where a mass loss equivalent to approximately 55 % loss of plasticiser occurs and it follows a kinetic rate law of the form

$$\ln (1-F) = -k_\alpha\, t \qquad \text{[Eq. 1C]}$$

where F is the fractional loss of plasticiser, t the time and k_α the appropriate 1st order rate constant.

This initial process then gradually gives way to a second mass-loss process of the form

$$\ln (1-F) = \gamma - k_\beta\, t \qquad \text{[Eq. 2C]}$$

where γ is curve fitting constant and k_β is the rate constant which is appropriate to the reaction.

Overall, by comparing these two sets of results, it can be seen that the larger the sample, the smaller the relative loss of plasticiser from the PBX in a given time.

The loss of plasticiser is accompanied with a change in surface hardness, although quantitatively, it appears more difficult to directly correlate changes in Shore A hardness with the loss of plasticiser. Initially, the Shore A hardness of both samples was 59 ± 3 Shore A units (10 measurements, top surface). After the 6 months accelerated ageing exposure, the hardness of the larger cylinder of PBXW-115 (Aust), Sample A, increased to 62 ± 3 and that of the smaller Sample B increased to 63 ± 3, differences which are not statistically significant.

In this context, it can be anticipated that, if a warhead filled with PBXW-115 (Aust.) is to be subjected to either accelerated ageing or a high temperature storage regime for a long period, it will be expected to shed some plasticiser and, most likely, the IDP plasticiser will just evaporate harmlessly into the atmosphere and eventually hydrolyse [22].

However, such fills are confined within a warhead case and, as a consequence, the relative mass loss will be expected to be less than that for unconfined samples. In addition, because the larger the PBX fill, the smaller the relative loss would be per unit time, one would expect that the change in mechanical properties with plasticiser loss over a given period would be much less than that for a small laboratory test sample, albeit one approximately 7 mm thick, spread out on a tray.

5.2 Changes in the Tensile Properties of PBXW-115 (Aust.)

In Table 3, a summary of the results from tensile testing carried out at low strain rate, at room temperature (20-25°C) of samples from PBXW-115 (Aust.), stored at laboratory temperatures in an airtight container over a period of twelve months is given.

These results indicate that, within the limits of the statistics, there have only been small changes in these PBXW-115 (Aust.) samples on ageing at room temperature in the laboratory in an airtight container.

Initially, the load bearing capacity, as measured by the maximum stress, increases during the first six months, from 0.48 MPa to 0.60 MPa; this probably occurs because increased cross-linking between residual, remotely-located or isolated pendant hydroxyl or isocyanate groups in the cured binder can take place [3, pp17-19] and this increases the ability of the polymer chains to carry an additional load. In addition, the extensibility of the material, as measured by the extension or strain at maximum load, appears to fall somewhat during the first three months, from 9.8 % to 8.8 %, before stabilising in the next three month period with a value ca. 10.7 %; again, this can be explained by assuming that reactions occur between remotely located molecular functionalities but the rate at which these reactions occur is particularly slow.

5.3 The Tensile Properties of PBXW-115 (Aust.), PBXN-103, PBXN-105 and PBXN-106

These results on the tensile properties of fresh PBXW-115 (Aust.) from Table 3 can be compared with those from Composition B [23], Composition H-6 [23], PBXN-111 [15], PBXN-103 [13-15, 21] and the closely related PBXs, PBXN-105 [13, 21] and PBXN-106 [13, 15] (Table 6).

Table 6: The Tensile Properties of PBXW-115 (Aust.), PBXN-111, PBXN-103, PBXN-105 and PBXN-106 at 20 - 25°C.

Material	Reference	Maximum Stress (MPa)	Strain (%) at Maximum Stress	Tangential Modulus (MPa)
PBXW-115 (Aust.)	This Report	0.484	9.76	6.18
PBXN-111	15	0.579	8.36	approx. 7
PBXN-103	15	0.848	14	7.9
	21	0.45 - 0.72	11.3	5
	13	0.45	12	5
	14	(cut) >0.40, (cast) >0.62	≥ 9	(cut) > 4.4, (cast) > 7
PBXN-105	24	1.11	8.2	18.5
	15	0.64	14.9	5.24
	13	1.23	13	11
PBXN-106	13, 15	0.32	10	6
Compare: Composition B	12, 23	1.43	0.02	11700
Composition H-6	13, 21, 23	2.63	0.02	12300

The tensile properties of Composition B and Composition H-6 [13, 23] are quite similar; they are relatively hard, brittle materials. They each have a relatively low tensile strength (as measured by the maximum stress data) but a very low elongation or extensibility (as measured by the strain data), resulting in a relatively high tangential modulus, characteristic of brittle materials. On the other hand, the tensile properties of PBXW-115 (Aust.), PBXN-111, PBXN-103 and PBXN-105 indicate that while they also have very low relative tensile strengths, they have correspondingly very high elongations or extensibilities, characteristic of the rubbery materials they were formulated to be and, as a result, these formulations behave as insensitive explosives (reduced sensitivity to fragment impact, mechanical drop and high compression impact) [21].

Mechanical properties do, however, alter with temperature (especially when one passes through the glass transition temperature) and the effect of this can be seen in the changes in tensile properties of PBXN-103 (1972 formulation, then known as Nitronal) [25], PBXN-105 [13, 15, 24] and PBXN-106 [15] (Table 7). The net result of this is that, if possible, one should determine mechanical properties of an explosive formulation over a range of temperatures, if one is going to be involved in operational work at low temperatures. Unfortunately, in the screening program undertaken at AMRL, Melbourne on PBXW-115 (Aust.), it was not possible to obtain data at other than at ambient laboratory temperatures.

Table 7: *The Temperature Dependence of the Tensile Properties,* σ_m, *Maximum Stress (MPa) /* ε_m, *Strain at Maximum Load (%) / E, Tangential Modulus (MPa)*

Temperature (°C)	-52	-40	20-25	60
	σ_m, (MPa) / ε_m, (%) / E, (MPa)	σ_m, (MPa) / ε_m, (%) / E, (MPa)	σ_m, (MPa) / ε_m, (%) / E, (MPa)	σ_m, (MPa) / ε_m, (%) / E, (MPa)
PBXN-103			0.79 / 29 / 3.27	0.25 / 22 / 1.45
PBXN-105		7.86 / 6.8 / 228	1.109 / 8.2 / 18.5	0.846 / 9.2 / 11.5
PBXN-106	6-9 / 0.6-1.1 / 86		0.24 - 0.32 / 10 / 6	0.18 / 17 / 3 (70°C)

5.4 Changes in the Tensile Properties of PBXW-115 (Aust.), PBXN-103 and PBXN-105 subjected to Accelerated Ageing

In Table 4, a summary of the experimental data from samples of PBXW-115 (Aust.) exposed to accelerated ageing under the Cycle B2 regime for a period of 12 months is presented.

From these results, it is apparent that there has been no major change in the tensile properties of PBXW-115 (Aust.), even after 50 weeks' exposure. Those changes which have occurred are best identified by comparing the results from accelerated aged samples (Table 4) with those from the controls which had been aged under ambient conditions (Table 3). The load bearing characteristics and the extensibilities of those samples aged at room temperature are slightly better than those of the accelerated aged materials. However, in neither set of samples does it appear as if any catastrophic change has occurred.

These results compare with those reported recently from PBXN-105, stored at 60°C for twelve months (Table 8, based on Table 4 from [24]). Here, it was found that there was very little change in the tensile properties at 25°C of PBXN-105 of UK manufacture over the first six months; however, after 12 months at 60°C, its load bearing properties, as measured by the maximum stress parameter, had fallen some 30 % and this was attributable primarily to the (unspecified) loss of an apparently critical mass of the energetic plasticiser, which in turn reflected itself in an abrupt, sharp increase in the glass transition temperature from -40°C to -20°C.

Actually, the behaviour of PBXN-105 is similar to that reported for PBXN-103 (1972 composition [25]), (Table 9). Here, it was found, after three months at 60°C, that the load bearing properties of PBXN-103 at 25°C had fallen approximately 25 %, even though its extensibility and tangential modulus were almost unchanged.

Table 8: Room Temperature (25 °C) Tensile Testing of PBXN-105 manufactured at Royal Ordnance Factory, Glascoed, UK and subjected to Storage at 60 °C for Periods up to One Year [24].

Sample	Maximum Stress, (N mm^{-2}), (MPa)	Strain at Max. Load (%)	Strain at Break, (%)	Modulus of Elasticity, (N mm^{-2}), (MPa)
Fresh Sample, zero time at 60°C	1.109	8.2	8.6	18.5
Aged Sample, 3 months at 60°C	1.170	9.8	10.4	18.0
Aged Sample, 6 months at 60°C	1.279	8.6	9.1	19.9
Aged Sample, 12 months at 60°C	0.726	7.6	8.9	12.5

Table 9: The Effect of Accelerated Ageing on an Early Formulation of PBXN-103, formerly known as Nitronal [25]

Exposure Tensile Test at	Control, 25°C 25°C	Control, 25°C 60°C	3 mo., 60°C 25°C	3 mo., 60°C 60°C
σ_m, Max. Stress (MPa)	0.799	0.248	0.586	0.220
ε_m, Elongation at max. stress (%)	25	19	23	15
ε_b, Elongation to Break, (%)	29	22	27	19
Initial Modulus, E (MPa)	3.27	1.45	3.10	1.45

While these changes may not be not critical (because there is no detail given as to the mass and dimensions of the samples being exposed or the mass losses involved), these observations still reinforce the need for ongoing surveillance of warheads filled with PBXs where they are subjected to long-term storage in a hot climate.

6. Commentary and Future Recommendations

6.1 The Interpretation of Accelerated Ageing Data and Recommendations for the Future

From the mass-loss study of PBXW-115 (Aust.) exposed to accelerated ageing under the B2 Cycle program, it is apparent that the loss of mass depends on the surface area of the exposed sample - the greater the surface area, the greater the mass loss. However, it is also apparent that the relative loss of mass per unit time is inversely proportional to some power of the cross-sectional area and/or the volume of the sample. In other words, the greater the cross-sectional area and the volume of the sample, the greater the loss of mass from the sample but the smaller the relative loss of mass would be. This can be seen by inspection of the data in Table 5 and from the analysis of that data in the form of Eqs. 1A and 1B and Eqs. 2A and 2B.

When small samples are investigated, one is looking for changes which may occur in a "worst case" or "very serious" scenario on samples which will be prone to the greatest change. One is looking to assess changes resulting from an exaggerated set of hostile conditions. When those experiments have been done, one is in a position to extend those results to larger stores.

Changes in the tensile properties of small samples of PBXW-115 (Aust.) have been shown to occur on ageing (Tables 3 and 4); however, the observed magnitude of the changes on the samples studied here is not very pronounced - the material has not become brittle or powdery and it is still rubbery as would be expected from a material formulated with a HTPB-based polyurethane binder, even though more than 60 % of the plasticiser has been lost (Table 5). This indicates that larger samples, for example those in a warhead store, exposed to similar external environments should retain their rubber-like properties and their structural integrity and, therefore, their operational safety and effectiveness.

What would happen if little or no plasticiser were present in the mix? Just what is the function of the plasticiser in PBXW-115 (Aust.)?

Again, these questions have not been specifically addressed and reported on in the studies on polyurethane-based PBXs. In the case of PBXW-115 (Aust.), at the very least, the plasticiser functions as a processing aid in the manufacturing of the PBX; it reduces the viscosity of the mix and enhances the efficiency of the mixing process and

the subsequent casting process. It probably has an additional role, functioning as a true plasticiser, reducing intermolecular interactions within the curing and the cured PBX matrix, improving its elasticity, lowering the glass transition temperature of the cured PBX and probably reducing its sensitivity as an explosive to impact and shock.

The rubber-like properties of PBXW-115 (Aust.), PBXN-111, PBXN-103 and PBXN-105 (Tables 6-9) are all enhanced by the presence of the plasticiser in their respective formulations [19] and, were the PBXs to be made up without plasticiser, processing would definitely become more difficult, the long-term storage properties may be adversely affected and explosive safety margins reduced.

Seen in this light and in the light of the 1990/91 surveillance program on PBXN-105 carried out at the Royal Armament Research and Development Establishment, the forerunner of the Defence Research Agency at Fort Halstead, Kent for the UK Ordnance Board [24], it is apparent that, in future, any on-going surveillance of PBX-filled ordnance should be planned in detail at the outset of any production plan. This will ensure that changes in the physical and explosive properties of the fill can be monitored more fully and recommendations can be made with a greater degree of confidence than at present.

Such results would complement results such as reported in this paper.

In any surveillance program, the charges to be monitored over the lifetime of the store should be of sufficient size to be realistic; they should be placed in realistic generic containers and they should be stored in a magazine alongside the filled warheads or in one which reflects the most hostile environment that most of the warheads in service will experience.

For a charge such as PBXW-115 (Aust.), perhaps five right-cylindrical charges, mass 25 kg each, with a diameter and length each of 26 cm, confined in a mylar sheet-lined epoxy cylinder, would adequately meet the bill. Such a container could be easily opened, the charge slid out, sectioned and assessed and then returned to storage.

Samples would be analysed for RDX, AP and plasticiser, samples cut to carry out an assessment of the shock sensitivity (Large Scale Gap Test [26]) and any other tests or measurements deemed appropriate (impact sensitivity, temperature of ignition, differential scanning calorimetry, mechanical properties, density, mass loss, hardness, plasticiser content, etc. [4]). Such a surveillance program should be economical to implement and could run for over forty years as the material from any one surveillance charge would not be expected to be consumed in any one set of tests.

7. Acknowledgments

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20. ABSTRACT

When PBXW-115 (Aust.) is exposed to accelerated ageing according to the program set out in DEFAUST 5168: Cycle B2, mass loss (attributed to loss of plasticiser, isodecyl pelargonate) occurs, the Shore A hardness increases and the tensile properties alter somewhat.

There is a link between these changes and it is probably related to the plasticiser content of the samples. From a kinetic analysis, it appears as if there may be two consecutive mass loss processes operating in the accelerated ageing of PBXW-115 and each obeys a first order reaction kinetics profile; the first predominates up to the stage where a mass loss equivalent to ca. 55 % of plasticiser occurs, the second operates where the plasticiser content is low. However, in the time domain studied (one year), there is no evidence of catastrophic structural change (loss of integrity or propensity to crumble) in PBXW-115 (Aust.) under worst case conditions (test pieces of small volume and large relative surface area), suggesting that the explosive charge in warheads filled with PBXW-115 (Aust.) should remain intact over a long interval of time.

The tensile properties of PBXW-115 (Aust.) are similar to that of its US counterpart and not dissimilar to those of PBXN-103, PBXN-105 and PBXN-106; all are rubber-like materials, with lower relative tensile strengths at 20-25°C (σ_m, maximum stress values of 0.3 to 1.1 MPa), higher relative extensibilities (ε_m, strains at maximum load of 8 -15%) and very low tangential moduli (E, 5-20 MPa), compared to those of Composition B and H-6 (σ_m 1-3 MPa, ε_m 0.02%, E ca.12000 MPa).